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### **Characterization of Uranium and Mercury in High Level Waste Sludge**

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**Introduction:** Approximately 130 million L of High-Level Radioactive Waste (HLW) material, the waste product associated with the dissolution of spent fuel rods for the recovery of plutonium, resides in carbon steel subsurface tanks awaiting treatment at the Savannah River Site (SRS). Little information exists on actinide speciation in HLW. This information is applicable to criticality and to HLW tank decontamination and treatment. For example, if U is present as the metal, the criticality risk could be greater than if U were present as an oxidized U(VI) form such as a sodium uranyl hydroxide solid. We do not expect waste U to be present in the metallic form because the HLW is the result of spent fuel dissolution, which is an oxidizing process. However, some degree of undissolved metal may be present in the waste.

**Methods and Materials:** Our study used synchrotron-based X-ray absorption fine-structure spectroscopy (XAFS) and molecular modeling to characterize the chemistry of metals in HLW sludge. This report discusses our first measurements of metal speciation in HLW samples. We investigated the bonding environment of uranium (Tank 8F) and mercury (Tank 11H) in two sludge samples. Data collection was conducted using a 13-element Ge detector.

**Results:** Our study yields the following conclusions for these two samples. In the Tank 8F sample, which contains roughly 5% wt. U, most of the U is present as a U(VI) (oxyhydr)oxide precipitate, which contains U in the second coordination shell. Preliminary analyses indicate that about 5 % of the total U in the Tank 8F sludge sample exists as gamma-uranium metal. Although Tank 8F is rich in Fe oxides, we did not observe Fe in the local environment of the U suggesting that U is not sorbed or (co) precipitated with Fe oxides in the sludge. The Tank 11H sludge typically contains a few percent Hg. The Hg in the Tank 11H sludge sample that we examined is present as a Hg(II)-oxide. No metallic Hg forms were observed. WSRC-MS-2001-00726.